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(54) EPITAXIAL COMPOSITE AND METHOD OF MAKING

(71) We, NORTH AMERICAN ROCKWELL CORPORATION, a corporation organized and existing under the laws of the State of Delaware, United States of America, having its principal place of business at 2300, East Imperial Highway, El Segundo, California, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to an epitaxial composite comprising a thin film of single crystal Group III—V compound semiconductor such as GaP or GaAs_{1-x}P_x, Ga_{1-y}Al_yAs, Ga_{1-z}In_zAs, where x and y have values between zero and one, AlAs . . . ; InAs . . . and other solutions thereof on a single crystal, electrically insulating substrate. The invention also relates to a process for producing in situ the epitaxial composite by reaction of an appropriate alkyl compound of the Group III constituent of the semiconductor in an atmosphere containing one or more hydrides or alkyls of the Group V constituent.

As microelectronic technology improves, the search for better semiconductor materials becomes more intense. While past process technology has concentrated on the use of germanium and silicon for the formation of semiconductor devices, other semiconductor materials are becoming increasingly more important. Zinc-blend III—V semiconductor compounds and alloys are considered to be among the most versatile of all semiconductor materials. For example, varactors, transistors, microwave diodes, light-emitting diodes, injection lasers, bulk microwave power sources, negative resistance amplifiers, and bulk-effect integrated circuits are all possible with gallium arsenide.

A heteroepitaxial composite comprising a continuous film of single crystal III—V compound such as GaP or the alloys; e.g. GaAs_{1-x}P_x, Ga_{1-y}Al_yAs, on a monocrystalline, electrically insulating substrate would be

extremely valuable. For example, by carrying out on a composite of single crystal III—V compound on an electrically insulating substrate appropriate device fabrication steps such as diffusion, epitaxial growth while dopant is added to the growing layer, photolithography, and other device fabrication steps, and subsequently etching away a portion of the III—V compound to form independent islands of the semiconductor or device structure on the insulator, it is possible e.g., to manufacture very high density integrated circuits, while still achieving optimal electrical isolation between adjacent elements. Similarly, such an island configuration further permits electrical isolation between microwave or other components formed on adjacent islands of the semiconductor compound.

In addition, at certain temperature levels some of the electrically-insulating substrates; e.g., BeO, exhibit very good thermal conductivity. Thus, a III—V compound device; e.g., an amplifier, on a BeO substrate can be operated at a wider temperature range than a device fabricated in bulk III—V compound. In contrast, bulk III—V compounds as substrate materials, have a low thermal conductivity which results in heating of the device and hence an increase in the noise of the amplifier. Accordingly, by fabricating the device; e.g., an amplifier, of III—V compound on BeO the heat would be conducted away by the substrate, with concomitant decrease of noise. It is also important, when devices made from III—V material are in a high temperature environment, for the insulator to act as a heat sink and dissipate the heat either externally added or generated by the device.

The present invention provides a composite comprising a monocrystalline, electrically insulating substrate composed of chrysoberyl, gadolinium gallium garnet, beryllium oxide, thorium oxide, calcium fluoride, or barium fluoride, and a single crystal film of a Group III and Group V semiconductor compound other than GaAs and InP epitaxially disposed on said substrate, the Group III constituent

being one or more of Ga, Al and In, and the group V constituent being one or more of As, Sb and P.

The invention also provides a process for producing an epitaxial film of a group III—V semiconductor compound other than GaAs and InP disposed on a single crystal substrate of chrysoberyl, gadolinium gallium garnet, beryllium oxide, thorium oxide, calcium fluoride or barium fluoride, which process comprises, heating the substrate in a reactor and introducing into the reactor, one or more gaseous hydrides or alkyls of As, Sb and P as Group V constituent, and one or more gaseous alkyls of Ga, In or Al as Group III constituent of the semiconductor. The process of the present invention preferably utilizes a reaction chamber having only one high temperature zone, and permits production of single crystal films of very high purity. Furthermore, the preferred process, in the formation of semiconductors containing Ga, utilizes either trimethylgallium or triethylgallium, which compounds overcome the disadvantages of known techniques which do not employ gallium compounds that are gaseous at room temperature and atmospheric pressure.

Also, the process of formation of the semiconductor thin film of the invention offers the possibility of decreasing side reactions to provide significant reduction in the impurity level. Accordingly, a level of purity and quality may be attained whereby the single crystal films that are formed may be fabricated into "Gunn-effect" devices, for example.

The substrates have the following crystal structures: thorium oxide, and gadolinium gallium garnet, cubic structure; BeO, hexagonal structure; or chrysoberyl, orthorhombic structure.

In the process of the invention the alkyl compound of the Group III metal is reacted with the hydride or an alkyl of the Group V constituent of the semiconductor. In the preferred form of the process; for example, in the preparation of III—V gallium compounds, the Group III alkyl comprises trimethylgallium (TMG) or triethylgallium (TEG) and the hydride comprises arsine, phosphine or stibine to produce the appropriate compounds or alloys: GaP, GaAs_{1-x}Sb_x, GaAs_{1-x}P_x, . . . and GaP_{1-x}Sb_x. Where the Group III metal is aluminium or indium, trimethyl aluminium or triethylaluminium, or trimethyl indium or triethylindium can be employed as the alkyl compound of the Group III metal.

In a preferred embodiment, the substrate is positioned in a reactor and heated to within a temperature range of 650°C. to 800°C in an atmosphere of the Group V constituent provided by decomposition of the constituent hydride or alkyl. In the formation of semiconductors containing Ga, gaseous TMG or TEG then is introduced into the reactor to

form the Group III—V compound semiconductor epitaxially on the substrate.

The constituent compounds may be introduced in a carrier gas selected from hydrogen, nitrogen and the Group O gases, for example, helium and argon. The constituent compounds may be introduced into the reactor under reduced pressure.

Preferably an excess of the Group V hydride or alkyl is provided over that stoichiometrically required to form the film.

Multilayer thin films of different III—V semiconductor compounds or alloys such as AlAs, and Ga_{1-y}Al_yAs or multilayers of a III—V compound or compounds containing different type impurities which produce p or n doped layers may be produced.

In the accompanying drawings like numerals designated like parts in the several figures.

Figure 1 is a greatly enlarged, fragmentary perspective view, in partial section, of a heteroepitaxial composite in accordance with the preferred embodiment of the present invention.

Figure 2 is a simplified schematic diagram of an apparatus for illustrating the inventive process for producing an epitaxial III—V semiconductor thin film on a single crystal substrate.

Referring now to the drawings, and more particularly to Figure 1, there is shown a heteroepitaxial composite 10 in accordance with the present invention. Composite 10 comprises a substrate 11 of a single crystal electrically insulating material such as BeO, ThO₂, BaF₂ or CaF₂. Disposed atop surface 12 of substrate 11 is a thin film 13 of a monocrystalline Group III—V compound or alloy semiconductor. Preferably, thin film 13 comprises one of the gallium-V group such as gallium phosphide, or alloy compositions gallium arsenide-phosphide GaAs_{1-x}P_x or gallium arsenide-antimonide (GaAs_{1-x}Sb_x) or the like. Other groups are the aluminum-V compound or alloy semiconductors; e.g., AlAs, AlP, AlSb, Ga_{1-y}Al_yAs, Ga_{1-y}Al_ySb and the indium-V compound or alloy semiconductor-thin films; e.g., Ga_{1-y}In_yAs, or InAs. The film 13 may also be a multilayer structure of different semiconductor compounds, or alloys such as AlAs or Ga_{1-y}Al_yAs disposed on surface 12, or of the same material containing layers of n-type doped and p-type doped, one upon the other. (In the chemical formula for the alloy, x or y represents a value between zero and one.) Composite 10, the crystallographic characteristics of which are discussed hereinbelow, may be produced in accordance with the vapor phase deposition process now to be described.

Referring now to Figure 2, there is shown an apparatus useful for practicing the epitaxial deposition process. As shown therein, a vertical reactor 20 is provided with a pedestal 21 of a material, such as carbon covered by a film of

silicon carbide, which can be inductively heated; e.g., by using RF heating coil 22 and an appropriate RF power source not shown. Pedestal 21 is supported axially within reactor 20, positioned so as to be heated by coil 22, by means of a suitable support 23. It should be understood, however, that the pedestal 21 can be tilted to accommodate changes in reactor shape etc. and gas flow pattern. Vertical reactor 20 is provided with an inlet 20a and an outlet 20b, the latter being connected to an appropriate gas exhaust indicated by the legend gas exit in Figure 2. A carrier gas, typically hydrogen or helium, from tank 24 may be flowed through vertical reactor 20 by opening valves 25 and 26. As shown, the carrier, if hydrogen, may be purified by passage through a DEOXO units 27, molecular sieves 28, and a liquid nitrogen cold trap 29, all well known in the vapor deposition art. The gas flow rate from tank 24 to reactor 20 is measured by a flow meter 30.

The system shown in Figure 2 further comprises a pair of tanks 31 and 32 which contain respectively arsine and phosphine mixed in controlled volumetric amounts with hydrogen or an inert carrier gas. By opening valves 33 or 34 respectively, gaseous arsine or phosphine can be flowed through reactor 20. The flow rates of the arsine and phosphine are measured respectively by flow meters 35 and 36.

The metal alkyl compound used as a starting material in the process is contained in liquid form in an appropriate stainless steel bubbler 37. If desired, bubbler 37 may be provided with a bath (not shown) to maintain the liquid compound at a desired temperature. Carrier gas flow into bubbler 37 is controlled by a valve 38, the flow rate being monitored by an individually regulated flow meter 39. The gas line 40 between bubbler 37 and the vertical reactor 20 may be provided with a heating jacket (not shown) when necessary to maintain the compound within line 40 in the vapor phase.

EXAMPLE I

As an illustrative example of the inventive process, the following procedure may be used to produce an epitaxial thin film 13 containing gallium on a single crystal electrically insulating substrate 11 using trimethylgallium as one of the source materials.

First, the substrate is appropriately sliced so that its deposition surface 12 exposes a known crystallographic plane. Substrate 11 is positioned within vertical reactor 20 atop pedestal 21 with the deposition surface 12 facing gas inlet 20a. Initially, the carrier gas is flowed through the vertical reactor 20, by opening valves 25 and 26, to cleanse the system. A flow rate, typically on the order of 1.5 liters per minute, may be used.

The trimethylgallium (TMG) used as the

Group III alkyl compound for the deposition has the chemical formula $(\text{CH}_3)_3\text{Ga}$. TMG exhibits a boiling point of 55.7°C and a melting point of -15.8°C . Although TMG is a liquid at room temperature, better control of the vapor pressure may be obtained by maintaining the TMG in bubbler 37 at a temperature of 0°C . To accomplish this, bubbler 37 is immersed in an ice bath.

Substrate 11 is heated to a temperature of between 650°C and 800°C by means of RF induction coil 22.

Next, valve 34 and optionally valve 33 is opened to permit a flow of Group V hydride or hydrides from tank 32 or both tanks 31 and 32 into vertical reactor 20. Preferably, the flow rate of hydride is adjusted to introduce into reactor 20 an excess of hydride over that stoichiometrically required for the formation of the III-V compound.

Next, valves 38 and 41 are opened and the carrier gas is permitted to flow through bubbler 37, thereby introducing gaseous TMG into reactor 20. A typical range of flow rates for gas through the bubbler, as measured by flow meter 39, is from about 3.5cc per minute to about 120cc per minute.

The TMG reacts with the Group V hydride or hydrides to produce a single crystal gallium-containing III-V compound, in situ, atop heated substrate 11.

In an alternative procedure, the vapors of the metal alkyl(s) and the hydride(s) are mixed in the proper proportions required to produce the compound and/or alloy semiconductor and decomposed at the heated pedestal at one atmosphere pressure as may be provided by a carrier gas such as He or H_2 , or under a reduced pressure, as might be provided by the pure reactants without the use of a carrier gas.

The metal alkyls and the hydrides, which are not diluted by a carrier gas and are under their own vapor pressures, are mixed either before or in the reaction chamber, depending upon their mutual chemical reactivity at room temperature, and decomposed on the heated substrate. For example, the amounts of material to be reacted may be adequately controlled by flowmeters placed between the reactant materials and a vacuum environment in the reactor portion; for example, as provided by evacuation of the vertical reactor via the gas exit.

A similar technique is used employing triethylgallium (TEG) as the Ga source material. Triethylgallium has the chemical formula $(\text{C}_2\text{H}_5)_3\text{Ga}$, and exhibits a boiling point at 143°C . Since the boiling point of TEG is higher than that of TMG, sufficient vapor is better obtained if the TEG is kept in liquid form in bubbler 37 at room temperature or above. However, it often is desirable to heat line 40 between bubbler 37 and reactor

20 to maintain the TEG in the vapor phase as it is being transported to reactor 20.

As noted earlier, the Group V hydride initially is preferably introduced into vertical reactor 20 at a flow rate adjusted to provide an excess of the hydride over that stoichiometrically required. The TEG₂ then is introduced into the chamber in flowing carrier gas.

By employing phosphine as the Group V hydride process may be used to produce an epitaxial film of GaP.

The formation of the Group III—V alloys, for example, GaAs_{1-x}Sb_x, can be accomplished by mixing stibine (SbH₃) with AsH₃ in the appropriate proportions and reacting the gas mixture with TMG at the heated pedestal to form the GaAs_{1-x}Sb_x.

If desired, a III—V semiconductor or alloy epitaxial film may be doped during formation. To accomplish this, during film deposition a controlled amount of an appropriate dopant is introduced in the gas phase into reactor 20, via valve 42 and flow meter 43, from a container 44 (see Figure 2). If an n-type semiconductor film 13 is desired, selenium (Se) or sulphur (S), for example, may be introduced in the present process in the form of H₂Se or H₂S, which gaseous hydride would be contained in container 44 in pure or diluted form. In the reactor, the elevated temperature causes dissociation of the controlled amount of hydride to provide dopant for the thin film, and hydrogen. If an acceptor dopant is desired to provide a p-type semiconductor film 13, a suitable compound such as dimethylzinc, (CH₃)₂Zn, dimethylzinc (DEZ), (C₂H₅)₂Zn, or dimethylcadmium may be provided in container 45. Dissociation of these compounds provides a zinc or cadmium dopant, respectively, and an organic by-product which is exhausted from the system by the carrier gas flow.

A film 13 of mixed alloy; for example, GaAs_{1-x}P_x (where x may take any value between zero and one) also may be grown using the above process. In this example, both arsine and phosphine are introduced into vertical reactor 20 during the deposition, by opening both of valves 33 and 34. The arsenic/phosphorus ratio may be altered by controlling the relative proportions of arsine and phosphine introduced into the deposition chamber. The resultant film 13 in this example comprises a solid solution of gallium arsenide-phosphide, and is formed epitaxially atop substrate 11.

The process described above has been used to grow for example GaP, GaSb, GaAs_{1-x}P_x, GaAs_{1-x}Sb_x, AlAs, Ga_{1-x}Al_xAs and In As single crystal films 13 epitaxially on a number of single crystal electrically insulating substrates 11 selected from beryllium oxide (BeO), thorium oxide (ThO₂), gadolinium gallium garnet (Gd₃Ga₅O₁₂), chrysoberyl

(Al₂BeO₄), calcium fluoride (CaF₂), and barium fluoride (BaF₂). In the latter two cases, CaF₂ and BaF₂, flaking of the deposited films was noted when the thickness of the film was greater than 5 microns, possibly resulting because of the difference in coefficient of thermal expansion between these substrates and the noted semiconductors. However, with the other substrates listed, excellent large area thin films were achieved. In each case, epitaxy was verified by X-ray and reflection electron diffraction studies. In addition, photographs of the surface microstructure of thin film 13 helped to identify the films as single crystal.

In preferred composites, the substrate is chrysoberyl, gadolinium gallium garnet, calcium fluoride, or barium fluoride, and the single crystal film is GaP or Ga(As_{1-x}P_x) wherein x has a value between 0 and 1.

Where gadolinium gallium garnet is employed as the substrate, it is preferred that the garnet has the (211) surface exposed, and that the (111) plane of the single crystal film is parallel to the exposed plane of the garnet.

It will be understood that various modifications of the herein described process also are considered within the scope of the present invention. For example, after initial nucleation of the film 13, very small controlled amounts of gaseous AsCl₃ and/or HCl may be introduced into reaction chamber 20. If AsCl₃ is used, it dissociates to form HCl in the hydrogen atmosphere. The HCl tends preferentially to etch away or remove some defects in the growing III—V film, thereby providing a more defect-free film 13.

It has also been found beneficial to anneal a film after complete coverage has been achieved, as this removes many defects associated with the growth at the heterogeneous interface. Annealing after a thicker film has been grown has also been found to be beneficial in improving quality.

Further, while exemplary deposition temperatures have been set forth hereinabove, these are not to be considered limiting. For example, growth of the III—V film at deposition temperatures above 800°C may be employed, but there may be some sacrifice in growth rate and possibly film quality.

While the examples above have involved the growth of semiconductor films having As or P as the Group V constituent, the invention is not so limited. Thus as described, films having antimony as a Group V constituent also may be made using the inventive process; for example, by substituting stibine (SbH₃) for the arsine or phosphine as discussed supra. The high boiling point of Sb as compared to As or P requires much more careful control of the SbH₃ concentration in the growth process.

In another embodiment, the process may be used to produce Group III—V compound thin films wherein the Group III constituent com-

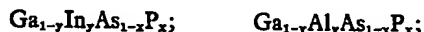
prises indium or aluminum. Thus; for example, an alkyl compound of In or Al may be introduced into reactor 20 in the gas phase. This alkyl compound reacts; e.g., with the hydride or arsenic, phosphorus or antimony to produce on heating InAs, InSb, AlAs, AlP and AlSb, for example. If the alkyl compound selected is one which reacts at room temperature with the hydride to form stable intermediate products, it may be desirable to use separate flow lines into reactor 20 for the alkyl compound and for the hydride. Preferably, at least one of the flowlines should terminate near the heated substrate.

15 EXAMPLE II

Preparation of Aluminum Arsenide (AlAs)

By substituting trimethylaluminum (TMA) for trimethylgallium (TMG) in the process described in Example I, and employing AsH₃ as the Group V hydride, a single crystal film of aluminum arsenide (AlAs) is produced on a single crystal electrically insulating substrate such as, beryllium oxide or chrysoberyl. An exemplary set of conditions for film growth is the following: temperature of pedestal, 700°C; AsH₃ (10%) in carrier gas (e.g., H₂ or He) at a flow rate of 90cc per minute into the reactor for about 2 minutes prior to introduction of TMA and then together with TMA carried by 50cc per minute of carrier gas which had been bubbled through the liquid TMA at room temperature. After 15 minutes of growth, approximately 4 μm of AlAs will have been grown, noticeably an orange film, transparent as seen through a transparent single crystal inorganic oxide substrate.

The process also may be used to produce Group III—V compound semiconductor thin films wherein the Group III constituent comprises an alloy. For example, by introducing organic compounds of In and Ga, Al and Ga, or In and Al into reactor 20 together with a Group V hydride, alloy semiconductors such as Ga_{1-y}In_yAs; Ga_{1-y}In_yP; Ga_{1-y}Al_yP; Ga_{1-y}Al_yAs; In_{1-y}Al_yP; In_{1-y}Al_yAs or the like will form on reaction at an elevated temperature. (In each case, y assumes a value between zero and one.) Of course, such systems also can be combined with mixtures of the Group V constituents to form such alloys as:



or the like or (GaAlIn)_a(AsPSB)_b, where a and b=1.

55 EXAMPLE III

Preparation of Ga_xAl_{1-x}As, an example of Alloy Formation

Of the mixed III—V alloys, Ga_xAl_{1-x}As can be prepared by reacting AsH₃ with mixtures of TMG and TMA. The composition

of the mixture can be changed by adjusting the rates of flow of the carrier gas through the liquid alkyl compounds and then mixing the gases prior to their introduction into the reactor, where reaction with arsine takes place to produce the alloy. The alloy formed is graded in composition; for example, by varying the TMG or TMA concentration during film growth by adjusting the carrier gas flows.

An alternate process for alloy growth of a desired composite would be to mix appropriate amounts of the liquid TMA and TMG together in a bubbler and use a carrier gas such as H₂ or He to transfer predetermined amount of the alkyls to the reaction zone.

As an alternative to use of the hydride to supply the Group V constituent of the compound semiconductor, in some instances an alkyl compound of the Group V constituent may be used. Examples of such a compound include trimethylstibine, (CH₃)₃Sb, and trimethylarsine, (CH₃)₃As, and trimethylphosphine, (CH₃)₃P.

The exemplary set of conditions for alloy growth by the alternate process is the following: pedestal temperature, 700°C; total carrier gas flow 1.5 liters/minute; carrier flow through TMG at 0°C, 10cc per minute; carrier flow through TMA of 15cc per minute; and flow of AsH₃ (10% in carrier) at a rate of 90 cc per minute in the reactor during growth and also about 2 minutes before introduction of the TMG and TMA into the reactor. These growth conditions produced a single crystal alloy with composition Ga_xAl_{1-x}As on the single crystal electrically insulating substrate.

WHAT WE CLAIM IS:—

1. A composite comprising a monocrystalline, electrically insulating substrate composed of chrysoberyl, gadolinium gallium garnet, beryllium oxide, thorium oxide, calcium fluoride or barium fluoride, and a single crystal film of a Group III and Group V semiconductor compound other than GaAs and In P epitaxially disposed on said substrate, the Group III constituent being one or more of Ga, Al and In, and the Group V constituent being one or more of As, Sb and P.

2. A composite according to claim 1 in which the semiconductor compound contains more than one of the said Group V elements.

3. A composite according to claim 1 or 2 in which the semiconductor compound contains more than one of the said Group III elements.

4. A composite according to any of Claims 1 to 3 in which the Group III—V semiconductor is impurity-doped.

5. A composite according to claim 1 in which the substrate is chrysoberyl, gadolinium gallium garnet, calcium fluoride or barium

fluoride and the single crystal film is GaP or Ga(As_{1-x}P_x) wherein x has a value between 0 and 1.

6. A composite according to any preceding claim in which the insulating substrate is gadolinium gallium garnet (Gd₃Ga₅O₁₂) having the (211) surface exposed, and the (111) plane of the single crystal film is parallel to the exposed plane of the garnet.

7. A process for producing an epitaxial film of a Group III—V semiconductor compound other than GaAs and In P disposed on a single crystal substrate of chrysoberyl, gadolinium gallium garnet, beryllium oxide, thorium oxide, calcium fluoride or barium fluoride, which process comprises, heating the substrate in a reactor and introducing into the reactor one or more gaseous hydrides or alkyls of As, Sb and P as Group V constituent, and one or more gaseous alkyls of Ga, In or Al as Group III constituent of the semiconductor.

8. A process according to claim 7 in which the constituent compounds are introduced under reduced pressure.

9. A process according to claim 7 or 8 in which the Group V constituent hydride or alkyl is introduced into the reactor before the alkyl compound of the Group III constituent.

10. A process according to claim 7, 8 or 9 in which the amount of the Group V constituent hydride or alkyl introduced exceeds that stoichiometrically required to form the said film.

11. A process according to any of Claims 7 to 10 in which alkyl compounds or more than one of the Group V constituents are introduced into the reactor.

12. A process according to any of claims 7

to 11 in which the Ga, Al and In alkyl compounds are trimethylgallium or triethylgallium; trimethylaluminium or triethylaluminium; trimethylindium or triethylindium.

13. A process according to any of claims 7 to 12 in which the concentration of the Group V constituent is controlled by adjusting the relative concentration of more than one of arsine, phosphine and stibine present in the reactor.

14. A process according to any of claims 7 to 13 in which the constituent compounds are introduced in flowing Group O gas.

15. A process according to claim 14 in which the Group O gas is helium or argon.

16. A process according to any of claims 7 to 13 in which the constituent compounds are introduced in flowing nitrogen.

17. A process according to any of claims 7 to 13 in which the constituent compounds are introduced in flowing hydrogen.

18. A process for producing an epitaxial composite substantially as described with reference to the accompanying drawings or in Example I, II or III herein.

19. A composite comprising a single crystal film of a III—V semiconductor compound epitaxially disposed on a monocrystalline electrically insulating substrate substantially as described in any of Example I, II or III herein.

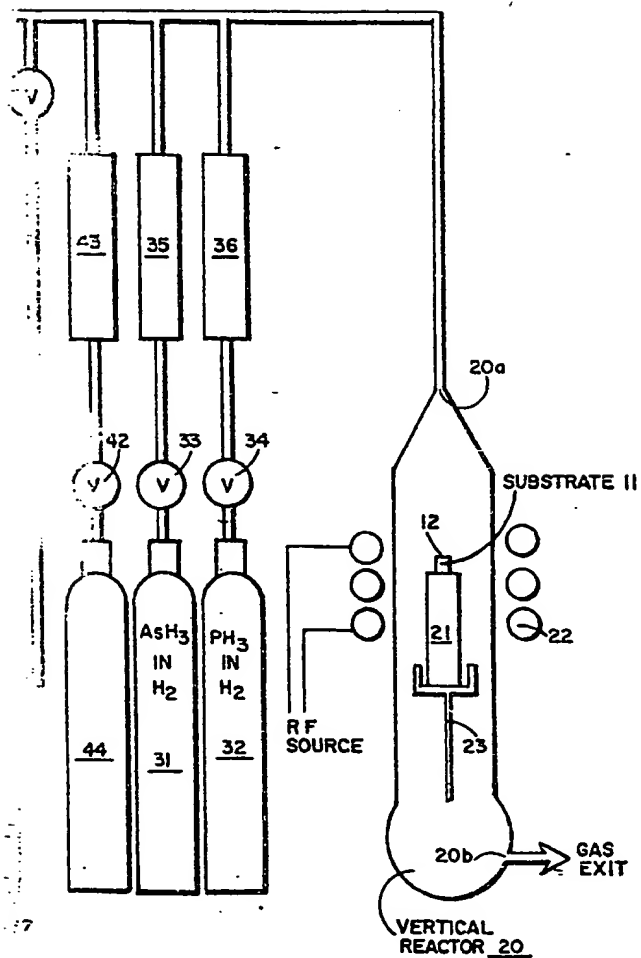
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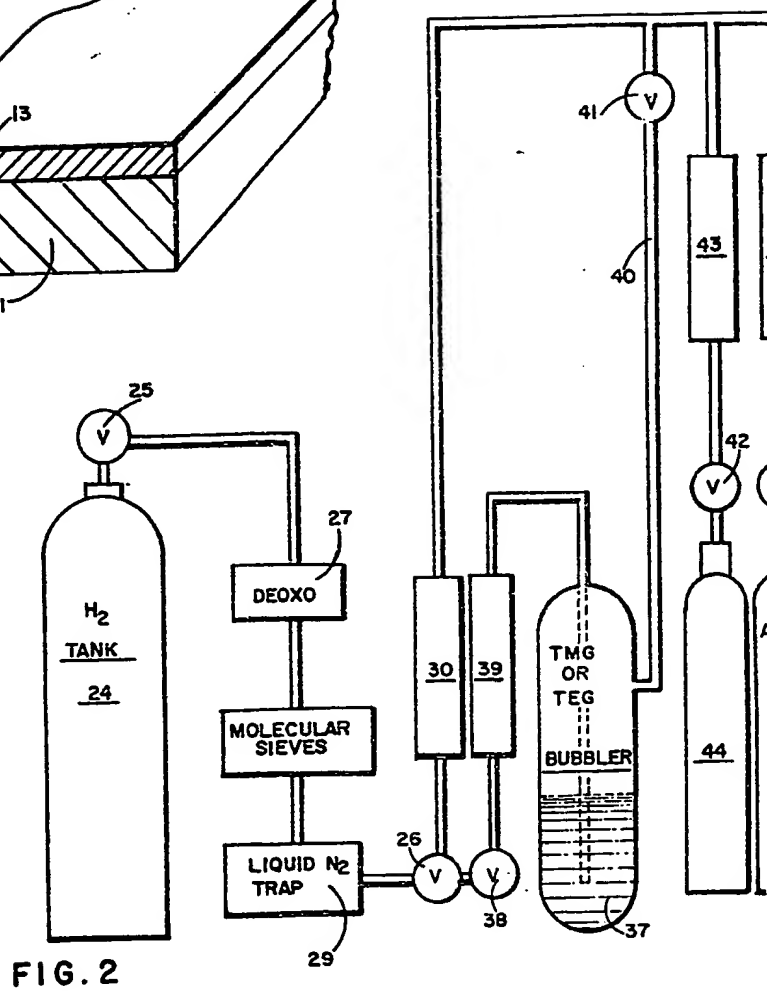
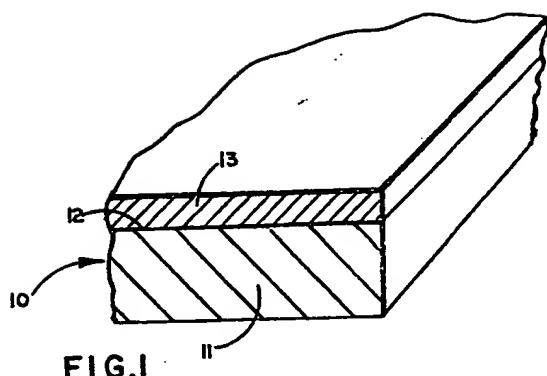
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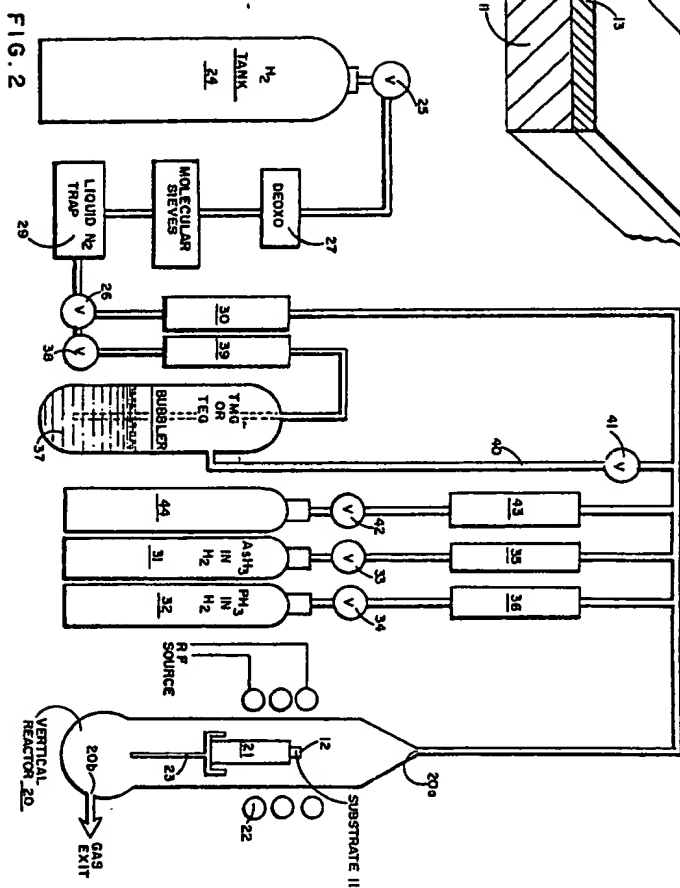
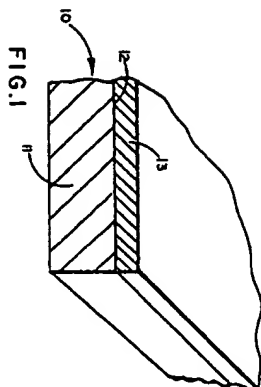
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